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Indian Standard

SPECIFICATION FOR REFINED CRESYLIC ACID

(First Revision)

Second Reprint APRIL 1992

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5 *December* 1968

Indian Standard

SPECIFICATION FOR REFINED CRESYLIC ACID

(First Revision)

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Indian Standard

SPECIFICATION FOR REFINED CRESYLIC ACID

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 17 July 1968, after the draft finalized by the Coal Carbonization Products Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first issued in 1956. It was felt by the Sectional Committee responsible for the preparation of the standard that this standard should be brought up to date with the trade practices in this country. In this revision the maximum limits of pyridine basis and neutral oils have been lowered. Sampling plan and criteria for conformity have also been revised.

0.3 This standard is one of a series of Indian Standards on coal carbonization products.

0.4 In the preparation of this revision, assistance has been derived from the following publications:

Standard methods of testing tar and its products. 1962. Standardization of Tar Products Test Committee, London.

BS 524 : 1964 Specification for refined cresylic acid. British Standards Institution.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for refined cresylic acid.

---

*Rules for rounding off numerical values (*revised*).

2. TERMINOLOGY

2.1 For the purpose of this standard, the following definitions shall apply.

2.2 Dry Point — The temperature at which the liquid just disappears from the bottom of the flask. The flame should be removed or extinguished immediately when this occurs, otherwise too high a temperature will be read. The dry point is affected by any superheating of the vapours which may occur and therefore cannot be recommended as an indication of the presence of high-boiling constituents.

2.3 Pitching Point — The point at which the temperature ceases to rise and begins to fall, while there is a residue in the flask.

3. GRADES

3.1 There shall be following three grades of the material:

Grade 1,

Grade 2, and

Grade 3.

3.1.1 The requirements of these grades are prescribed in Table 1.

TABLE 1 REQUIREMENTS OF GRADES OF REFINED CRESYLIC ACID

(*Clauses* 3.1.1, 4.5 *and* 4.6)

GRADE	MAXIMUM PERCENT OF				
	Water (v/v) W	Pyridine Bases (w/v) (B)	Neutral Oils (v/v) (N)	($B+N$)	($W+B+N$)
(1)	(2)	(3)	(4)	(5)	(6)
1	*See* col 5 and 6	0·25	0·2	0·4	1·0
2		1·5	1·5	2·5	3·0
3		2·0	2·5	4·0	5·0

4. REQUIREMENTS

4.1 Description — The material shall be a mixture of monohydric phenols consisting essentially of cresols with or without xylenols and with or without a small proportion of phenol.

4.2 Colour — The colour of the material shall be one of the following shades as agreed to between the purchaser and the supplier:

a) White,
b) Very pale,
c) Pale,
d) Medium dark, and
e) Dark.

4.2.1 The colour of the material shall be tested by the method prescribed in Appendix A.

NOTE — The colour of cresylic acid tends to darken on keeping and on exposure to light.

4.3 Distillation Range — The distillation range of the material shall be as agreed to between the purchaser and the supplier, and the test shall be carried out by the method prescribed in *IS : 1448 [P : 18]-1967.

4.4 Residue on Distillation — The material shall be free from pitch when tested by the method prescribed in Appendix B.

4.5 Water — The material shall not contain more than the percentages of water given in Table 1, when tested by the method prescribed in †IS : 1448 [P : 40]-1967 using a 2-ml receiver and solvent naphtha (boiling range 125° to 160°C) as the carrier liquid.

4.6 Pyridine Bases and Neutral Oils — The material shall not contain more than the percentages of pyridine bases and neutral oils given in Table 1, when tested by the method prescribed in Appendix C.

4.7 Freedom from Acids and Alkalis — The material shall not cause any change in colour of the phenol red indicator when tested in accordance with **4.7.1**.

4.7.1 Shake the sample thoroughly. Take one drop on a glass rod and add to three drops of the phenol red indicator on a tile. Mix well and note the colour.

4.8 Hydrogen Sulphide — The material shall impart not more than a pale yellow colouration to lead acetate paper when tested in accordance with **4.8.1**.

4.8.1 Take 20 ml of the material in a 100 ml conical flask. Place a piece of filter paper, freshly moistened with a 10 percent (*w/v*) aqueous solution of lead acetate, over the mouth of the conical flask. Place the flask on a boiling water bath. Examine the paper after five minutes.

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*Methods of test for petroleum and its products : Distillation (*first revision*).

†Methods of test for petroleum and its products : Water by distillation (*first revision*).

5. QUALITY OF REAGENTS

5.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis.

6. SAMPLING

6.1 Representative samples of the material, not less than 500 ml, shall be drawn as prescribed in Appendix D.

7. PACKING AND MARKING

7.1 Packing — The material shall be packed in amber coloured or dark blue glass bottles fitted with ground glass stopper or as agreed to between the purchaser and the supplier.

7.2 Marking

7.2.1 Each container shall be securely closed and marked with the name of the manufacturer; weight of the material in the container; recognized trade-mark, if any, and the year of manufacture.

7.2.2 Each container shall also have the caution label 'POISON! PROTECT FROM LIGHT AND MOISTURE' together with the corresponding symbol for labelling of dangerous goods as given in Fig. 5 of IS : 1260-1958†.

7.2.3 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

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*Specification for water, distilled quality (*revised*).

†Code of symbols for labelling of dangerous goods.

APPENDIX A

(*Clause* 4.2.1)

DETERMINATION OF DEPTH OF COLOUR

A-0. GENERAL

A-0.1 Outline of the Method — The colour of the sample is compared against standard solutions by viewing equal depths in the Nessler cylinders.

A-1. APPARATUS

A-1.1 Nessler Cylinders — Two flat-bottomed cylinders of thin colourless glass about 25 mm in diameter and about 150 mm in length graduated at 50 ml. The depth measured internally from the graduation mark to the bottom shall not vary by more than 2 mm in the cylinders used for the test.

A-2. STANDARD COLOUR SOLUTIONS

A-2.1 The standard colour solutions shall contain per litre the following quantities of solutes of analytical reagent quality, dissolved in distilled water:

Colour of Solution		*Solute*	*Quantity of Solute* g
White		Cobalt sulphate	0·90
		Potassium dichromate	0·015
Very Pale	Red shade	Cobalt sulphate	6·0
		Potassium dichromate	0·015
	Yellow shade	Cobalt sulphate	4·0
		Potassium dichromate	0·075
		Copper sulphate	1·0
Pale	Red shade	Cobalt sulphate	22·5
		Potassium dichromate	0·06
	Yellow shade	Cobalt sulphate	10·0
		Potassium dichromate	0·18
		Copper sulphate	2·5
Medium Dark	Red shade	Cobalt sulphate	70·0
		Potassium dichromate	0·5
	Yellow shade	Potassium ferricyanide	320·0

A-2.1.1 The standard solutions except that containing potassium ferricyanide, keep well but may be used over a period not exceeding one month from preparation. The potassium ferricyanide solution shall be prepared on the day of the test.

A-3. PROCEDURE

A-3.1 Compare the sample with the standard solutions by viewing equal depths in the Nessler cylinders and report the colour and shade of the sample as not darker than that of the appropriate solution as typified by the description of the depth and shade of colour.

APPENDIX B

(*Clause* 4.4)

DETERMINATION OF RESIDUE ON DISTILLATION

B-1. PROCEDURE

B-1.1 The method prescribed in *IS: 1448 [P: 18]-1967 shall be used subject to the following modifications.

B-1.1.1 Use 100 g, instead of 100 ml of the well-mixed material, this being weighed into the already tared distillation flask.

B-1.1.2 When all the water has distilled over and the rate of distillation has increased to 3·0 to 3·5 ml per minute (about one drop per second), the distillation shall be continued, without intermediate measurements, until either the dry point or the pitching point is reached and then the flame shall be immediately extinguished.

B-1.2 When the flask has cooled, weigh the flask and contents and report the weight of the contents as residue on distillation.

B-1.3 The material shall be considered to be free from pitch, if the weight of the residue obtained is not more than 2 g.

APPENDIX C

(*Clause* 4.6)

DETERMINATION OF NEUTRAL OILS AND PYRIDINE BASES

C-1. APPARATUS

C-1.0 The following apparatus, which has been previously cleaned and made free from traces of grease, shall be assembled as shown in Fig. 1.

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*Methods of test for petroleum and its products : Distillation (*first revision*).

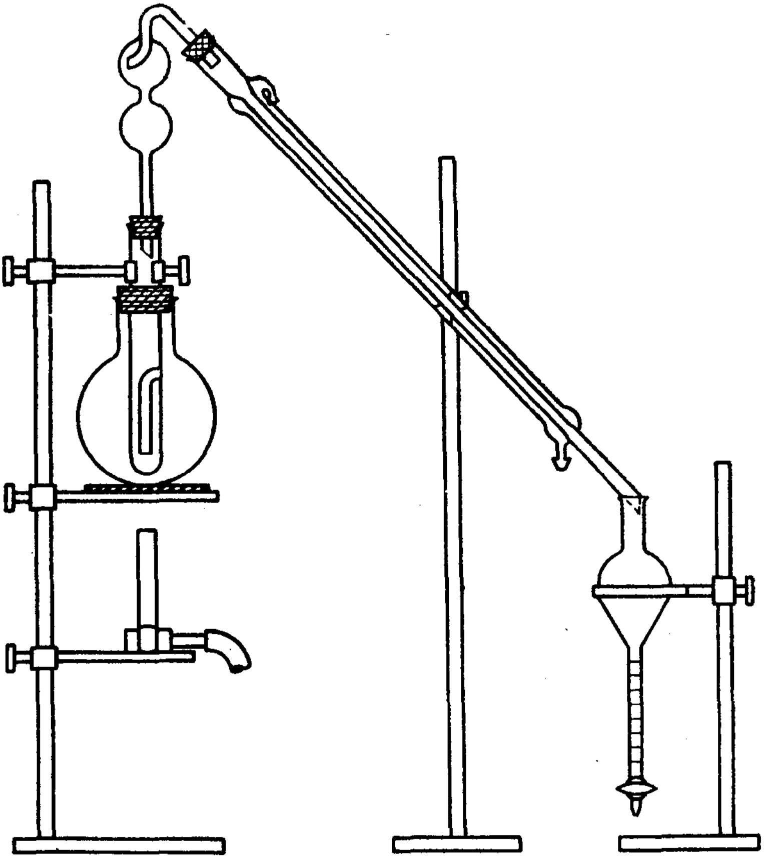
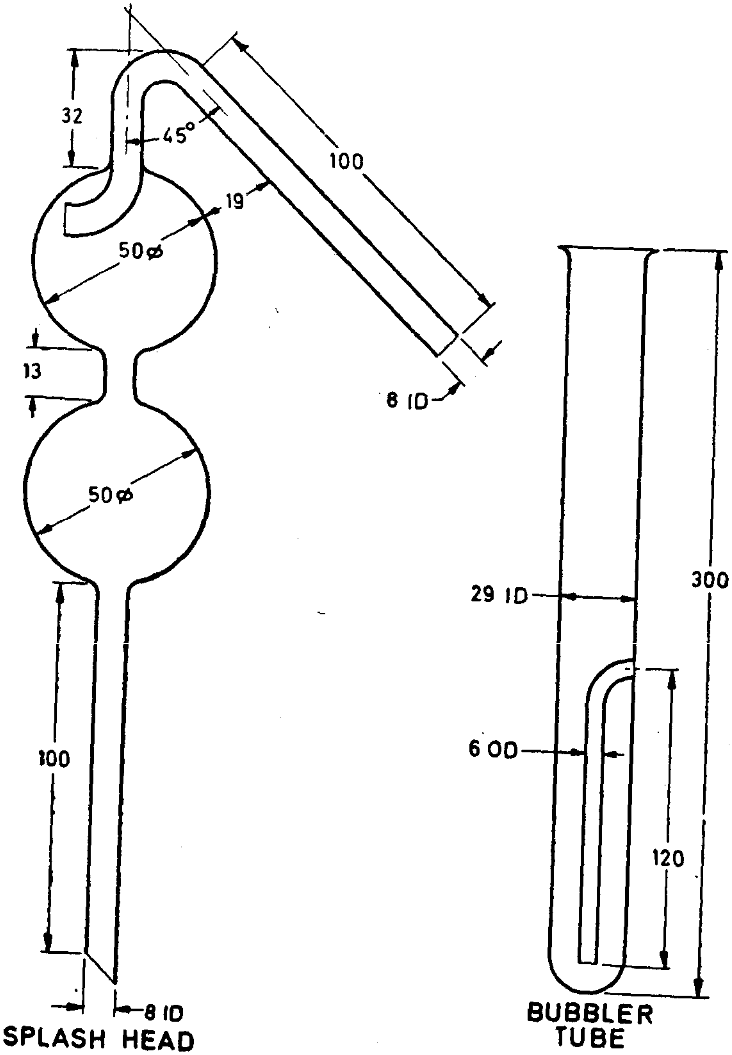


FIG. 1 APPARATUS FOR DETERMINATION OF NEUTRAL OILS AND PYRIDINE BASES

C-1.1 Flask — 1 000-ml capacity and of round bottom.

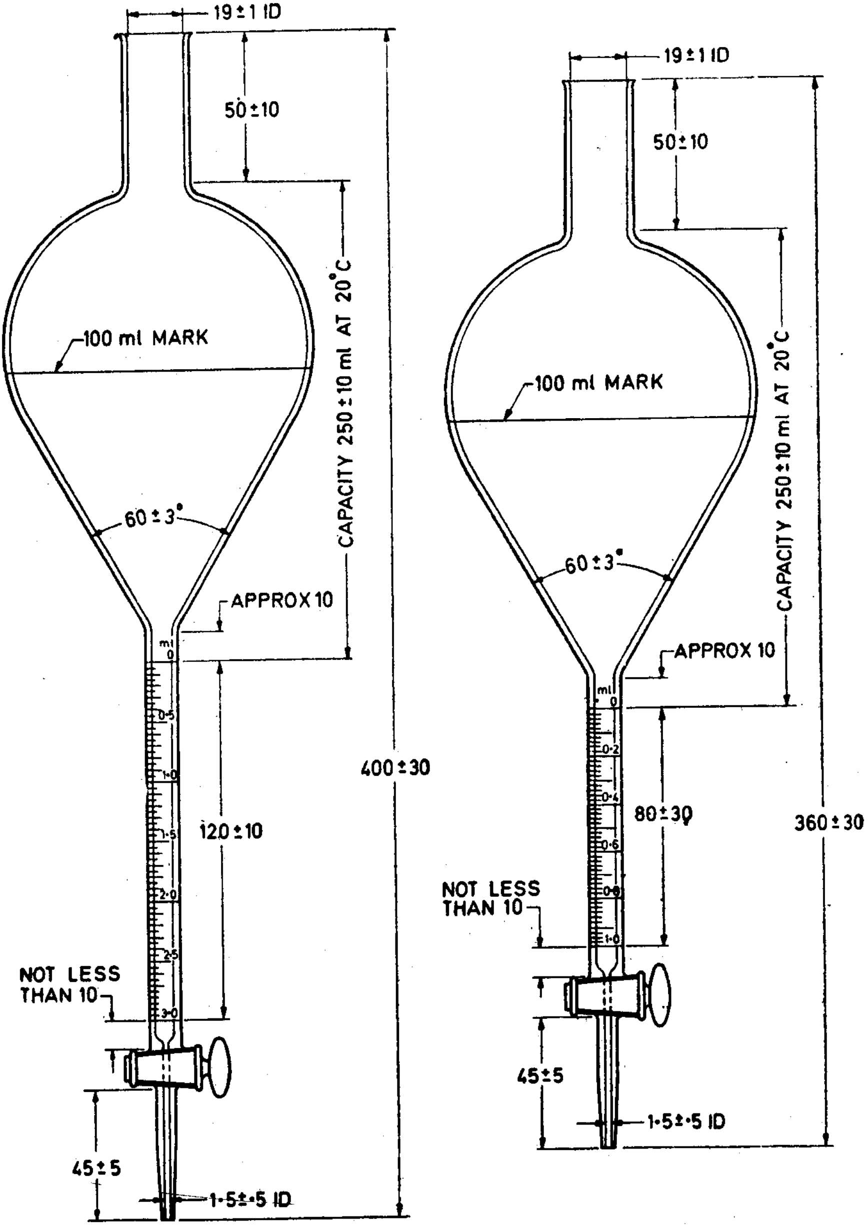
C-1.2 Splash Head and Bubbler Tube — of the shape and dimensions as shown in Fig. 2.

C-1.3 Graduated Separating Funnel Receivers — of the shape and dimensions as shown in Fig. 3. The bigger receiver shall be used for samples containing more than 1 percent neutral oil.



All dimensions in millimetres.

FIG. 2 DETAILS OF SPLASH HEAD AND BUBBLER TUBE



All dimensions in millimetres.

FIG. 3 SEPARATING FUNNEL RECEIVERS

C-2. REAGENTS

C-2.1 Sodium Hydroxide Solution — 27 percent by weight.

C-2.2 Sodium Chloride Solution — 2 percent by weight.

C-2.3 Phenolphthalein Indicator — Dissolve 0·1 g of phenolphthalein in 100 ml of 50 percent aqueous ethanol (or rectified spirit), which has previously been neutralized to the indicator.

C-2.4 Dilute Hydrochloric Acid — approximately 0·1 N.

C-2.5 Methyl Orange-Xylene Cyanol Indicator — Dissolve 1 g of methyl orange and 1·4 g of xylene cyanol FF in 500 ml of 50 percent (*v/v*) ethyl alcohol.

C-2.6 Standard Hydrochloric Acid — 1·0 N.

C-3. PROCEDURE

C-3.1 Before commencing the test it is essential to ensure that the receiver and conical flask are scrupulously clean. Washing with detergent, followed by thorough rinsing in tap water and distilled water, is usually adequate.

C-3.1.1 Measure 100 ml of the sample at laboratory temperature into the bolthead flask. Add 170 ml of the sodium hydroxide solution, followed by 100 ml of water using the same measuring cylinder, without intermediate cleaning or rinsing, for all three operations. Mix thoroughly. To prevent bumping add a small fragment of porous pot. Measure 25 ml of the sodium hydroxide solution into the inner bubbler tube. Fill the graduated portion of the receiver with the sodium chloride solution to prevent lodgement of the neutral oil, and assemble the apparatus as shown in Fig. 1.

C-3.1.2 Distill the material at such a rate that 100 ml of condensate is collected within 10-15 minutes. Ensure that the condensate runs down the inside surface of the receiver and not directly on to the liquid surface. Towards the end of the distillation turn of the water and drain approximately two-thirds of the condenser to melt any condensed solids. Do not allow steam to issue from the end of the condenser at any time during the distillation.

C-3.2 Neutral Oil — If necessary warm the distillate in the receiver sufficiently just to liquefy any solids. Run the aqueous distillate into the titration flask, cautiously, so that the neutral oil is contained in the graduated portion of the receiver. Note the volume of oil obtained and report as the percentage of neutral oil (*v/v*).

C-3.3 Pyridine Bases — Add the neutral oil in the receiver to the aqueous distillate in the conical flask. Wash the receiver with two successive quantities of 10 ml of water and transfer the washing to the conical flask. Add three drops of the phenolpathalein indicator. If a colour develops add 0·1 N hydrochloric acid solution dropwise from a burrette until the colour disappears. To this solution or, if no colour has developed, to the original solution, add two drops of the screened methyl orange-xylene cyanol indicator. Titrate with the 1·0 N hydrochloric acid solution and shake the mixture thoroughly after each addition of acid until the sharp colour-change of green to magenta is obtained.

C-4. CALCULATION

C-4.1 The percentage (w/v) of pyridine bases expressed as pyridine is 0·079 A where A is the volume, in millilitres, of 1·0 N hydrochloric acid solution added.

NOTE — It is desirable to clean the apparatus after each determination by distilling 400 ml of water in the assembled apparatus but without water flowing through the condenser.

APPENDIX D

(*Clause* 6.1)

SAMPLING OF REFINED CRESYLIC ACID

D-1. GENERAL REQUIREMENTS OF SAMPLING

D-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

D-1.1 Samples shall not be taken in an exposed place.

D-1.2 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

D-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution.

D-1.4 The samples shall be placed in suitable, clean, dry and air-tight glass containers, preferably of amber or blue colour.

D-1.5 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

D-1.6 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with full details of sampling, such as the date of sampling, the year of manufacture of the material, the batch number and the name of the sampler. Particular care shall be taken to ensure that sealing methods do not contaminate the sample.

D-1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

D-1.8 Phenolic substances burn the skin and may be absorbed into the system through the skin. It is essential for the sampler to wear gloves, preferably of polyvinyl chloride, and also a face shield. Inhalation of the vapours from hot material is to be avoided.

D-2. SAMPLING INSTRUMENT

D-2.0 The following forms of sampling instruments may be used:

a) Sampling bottle or can for taking samples from various depths in large tanks, and

b) Sampling tube.

D-2.1 Sampling Bottle or Can — It consists of a weighed bottle or metal container with removable stopper or top, to which is attached a light chain (Fig. 4). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

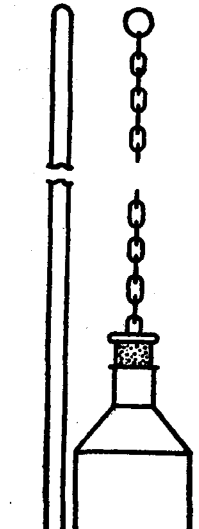


FIG. 4 SAMPLING BOTTLE OR CAN

D-2.2 Sampling Tube — It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (Fig. 5). The upper and lower ends are conical and reach 5 to 10 mm in diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

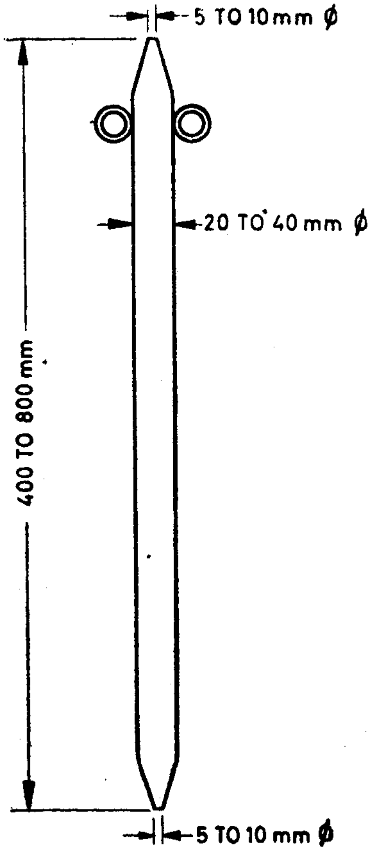


FIG. 5 SAMPLING TUBE

D-2.2.1 For small containers, the size of the sampling tube may be altered suitably.

D-3. SCALE OF SAMPLING

D-3.1 Lot — In a single consignment, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of containers of different sizes or of different batches of manufacture, then the containers belonging to the same size shall constitute a separate lot. In case the consignment is in large tanks or vessels, then the tanks or vessels belonging to the same batch of manufacture shall constitute a lot.

D-3.1.1 For ascertaining the conformity of the lot to the requirement of the specification, tests shall be carried out for each lot separately.

D-3.2 Sampling from Containers — The number (*n*) of containers to be selected for sampling shall depend on the size (*N*) of the lot and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE (*N*) (1)	NO. OF CONTAINERS TO BE SELECTED (*n*) (2)
Up to 100	5
101 ,, 200	6
201 ,, 300	7
301 ,, 400	8
401 ,, 500	9
Above 501	10

D-3.2.1 The containers shall be selected at random and to ensure the randomness or selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

> Starting from any container, count them in one order as 1, 2, 3 . . . ., etc, up to *r* and so on, where *r* is the integral part of N/n (*N* being the lot size and *n* being the number of containers to be selected). Every *r*th container thus counted shall be withdrawn to give sample for test.

D-3.3 Sampling from Tanks or Vessels — Each of the tanks or vessels in the lot shall be sampled separately for determining the conformity of the lot of the requirements of the specification.

D-4. PREPARATION OF TEST SAMPLES

D-4.1 Test Samples from Containers — To ensure that the sample taken from each container is fairly representative, the contents shall be mixed thoroughly, when possible by shaking or stirring or rolling. Draw small samples of the material from various depths with the help of the sampling tube (Fig. 4). The approximate quantity of the materials to be drawn from a container shall be a little more than thrice the quantity required for testing purposes as indicated in **D-5.1** and **D-5.2**.

D-4.1.1 Out of the material drawn from each individual container, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determination for all the characteristics specified under **D-5.2.** The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

D-4.1.2 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers selected shall be for the purchaser, another for the supplier, and third for the referee.

D-4.1.3 All the individual and composite samples shall be transferred to separate sample containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **D-1.6.**

D-4.1.4 The referee test samples, consisting of a composite sample and a set of *n* individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two, to be used in case of any dispute.

D-4.2 Test Samples from Tanks or Vessels

D-4.2.1 For drawing a sample from a tank or vessel, lower the closed sampling bottle or can (**D-2.1**) slowly to the required depth, open and fill it at that depth. Three samples shall be obtained at levels of one-tenth of the depth of the liquid from the top surface (top sample), one-half of the depth (middle sample) and nine-tenths of the depths of the liquid from the top surface (lower sample). All the three samples thus obtained from a tank or vessel shall be mixed together in a clean dry container and shall be divided into three parts, one for the purchaser, another for the supplier and the third for the referee. Each of the tanks or vessels in the lot shall be sampled in the above manner and separate samples obtained for each of the tanks or vessels. The approximate quantity of the material to be drawn from a tank or vessel shall be a little more than thrice the quantity required for carrying out tests for all the requirements prescribed in **D-5.1** and **D-5.2.**

D-4.2.2 All the samples thus obtained from tanks or vessels in the lot shall be transferred to separate sample containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **D-1.6.**

D-4.2.3 The referee test samples, consisting of the samples from the tanks or vessels in the lot, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two, to be used in case of any dispute.

D-5. NUMBER OF TESTS

D-5.1 Tests for the determination of distillation range and residue on distillation shall be conducted on each of the individual samples (*see* **D-4.1.2**).

D-5.2 Tests for the determination of the remaining characteristics shall be conducted on the composite sample (*see* **D-4.1.1**).

D-6. CRITERIA FOR CONFORMITY

D-6.1 The lot shall be declared as conforming to the requirements of distillation range and residue on distillation if test results for each of the individual samples tested in respect of these characteristics satisfy the requirement as given in **4.3** and **4.4** respectively.

D-6.2 For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **D-5.2**) the test results for each of the characteristic shall satisfy the relevant requirements given in the specification.

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‡Peenya Industrial Area 1st Stage, Bangalore Tumkur Road BANGALORE 560058	38 49 55 38 49 56
Gangotri Complex, 5th Floor, Bhadbhada Road, T. T. Nagar, BHOPAL 462003	6 67 16
Plot No. 82/83, Lewis Road, BHUBANESHWAR 751002	5 36 27
53/5, Ward No. 29, R.G. Barua Road, 5th Byelane, GUWAHATI 781003	3 31 77
5-8-56C L. N. Gupta Marg (Nampally Station Road), HYDERABAD 500001	23 10 83
R14 Yudhister Marg, C Scheme, JAIPUR 302005	6 34 71 6 98 32
117/418 B Sarvodaya Nagar, KANPUR 208005	21 68 76 21 82 92
Patliputra Industrial Estate, PATNA 800013	6 23 05
T.C. No. 14/1421, University P.O., Palayam TRIVANDRUM 695035	6 21 04 6 21 17

Inspection Offices (With Sale Point):	
Pushpanjali, First Floor, 205-A West High Court Road, Shankar Nagar Square, NAGPUR 440010	2 51 71
Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005	5 24 35

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*Sales Office in Calcutta is at 5 Chowringhee Approach, P. O. Princep Street, Calcutta 700072 27 68 00

†Sales Office in Bombay is at Novelty Chambers, Grant Road, Bombay 400007 89 65 28

‡Sales Office in Bangalore is at Unity Building, Narasimharaja Square, Bangalore 560002 22 36 71

Reprography Unit, BIS, New Delhi, India